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Indian Standard

SPECIFICATION FOR ACETYLENE BLACK

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BUREAU OF INDIAN STANDARDS
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Indian Standard

SPECIFICATION FOR ACETYLENE BLACK

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(Continued on page 2)

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(Continued on page 14)

Indian Standard

SPECIFICATION FOR ACETYLENE BLACK

0. FOREWORD

0.1 This Indian Standard was adopted by the Bureau of Indian Standards on 31 August 1987, after the draft finalized by the Inorganic Chemicals (Misc) Sectional Committee had been approved by the Chemical Division Council.

0.2 Acetylene black is a form of carbon with high electrical conductivity; made by decomposition by heat.

0.3 In present day dry cells, the cathode is manganese dioxide with about 10 - 30 percent of acetylene black to improve the matrix conductivity. The high carbon content in acetylene black (about 92 percent) makes it attractive for conversion to carbon.

0.4 In the preparation of this standard, considerable assistance has been derived from JIS K1469 - 1966 Acetylene Black. Two grades have been specified in this standard as per the requirements given in Table 1. These are known as 50 percent and 75 percent compressed material, respectively, in trade.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2 - 1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements, and methods of sampling and test for acetylene black for use in the manufacture of dry cells.

2. GRADES

2.1 Acetylene black shall be of two grades:

- a) Grade 1, and
- b) Grade 2.

*Rules for rounding off numerical values (revised).

3. REQUIREMENTS

3.1 Acetylene black shall be free from impurities which might have a deteriorating influence on the efficiency of the dry cells.

3.2 The material when tested according to the methods prescribed in Appendix A shall comply with the requirements given in Table 1. Reference to the relevant clauses of Apperdxix A is given in col 5 of Table 1.

TABLE 1 REQUIREMENTS FOR ACETYLENE BLACK

Sl No.	CHARACTERISTIC	REQUIREMENT		METHOD OF TEST REF TO CL NO. IN APPENDIX A
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Moisture, percent by mass, <i>Max</i>	0.4	0.4	A-2
ii)	Ash, percent by mass, <i>Max</i>	0.2	0.2	A-3
iii)	Coarse particles percent by mass, <i>Max</i>	0.04	0.04	A-4
iv)	Hydrochloric acid absorption number* (ml/5 g), <i>Min</i> or Acetone absorption number* (ml/5 g), <i>Min</i>	22.0	20.0	A-5
v)	Electrical resistivity (ohm-cm), <i>Max</i>	0.25	0.25	A-7
	Pour density† (g/ml)	0.05 to 0.09	0.09 to 0.11	A-8
vi)	or Apparent density† (g/ml)	0.08 to 0.10		A-9
	pH value	6-8	6-8	A-10
*Hydrochloric acid absorption number or acetone absorption number — one of these whichever is agreed upon between the purchaser and the supplier, only need to be taken as criterion for requirement in passing for standard marking.				
†Either of these to be tested as agreed upon between the purchaser and the supplier and to be taken as criterion for the requirement in passing for standard marking.				

4. PACKING AND MARKING

4.1 Packing — The product shall be packed in a suitable strong container as agreed to between the purchaser and the supplier.

4.2 Marking — Each package shall bear legibly and indelibly the following information:

- a) Name and grade of the material ;
- b) Name of the manufacturer and his recognized trade-mark, if any;
- c) Net mass;
- d) Date of packing ; and
- e) Lot number.

4.2.1 The package may also be marked with the Standard Mark.

NOTE—The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer, Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards.

5. SAMPLING

5.1 The method of drawing representatives samples of the material, number of tests to be performed and the criteria for finding the conformity of the material to the specification is prescribed in Appendix B.

APPENDIX A

(Clause 3.2)

METHODS OF TEST FOR ACETYLENE BLACK

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS : 1070 - 1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Specification for water for general laboratory use (second revision).

A-2. MOISTURE

A-2.1 One gram of acetylene black is weighed accurately into a weighing bottles (87 mm in outside diameter). Spread it on the bottom of the bottle in uniform thickness and dry for two hours in air oven maintained at 105 to 110°C. This is transferred to a desiccator and cooled to room temperature. It is weighed accurately and the moisture content calculated.

A-2.2 Calculation

$$\text{Moisture, percent by mass} = \frac{100 \, m}{M}$$

where

m = loss in mass in g on drying, and
 M = mass in g of the material taken for the test.

A-3. ASH

A-3.1 Two grams of acetylene black is weighed accurately into 50 ml silica crucible and heated gently in an electric furnace with the precaution against its scattering. Heating is continued at $750 \pm 10^\circ\text{C}$ for more than two and a half hours to attain constant mass and then allow to cool in a desiccator. It is weighed accurately and the ash is calculated.

A-3.2 Calculation

$$\text{Ash, percent by mass} = 100 \times \frac{M_1}{M}$$

where

M_1 = mass in g of the residue obtained, and
 M = mass in g of the material taken for the test.

A-4. COARSE PARTICLES

A-4.1 Apparatus

A-4.1.1 Test Sieve — 150 micron IS sieve (*see* IS : 460 - 1962*).

A-4.2 Procedure — 50 g of the sample is weighed accurately into a container of constant volume. Place it on the 150 micron IS sieve. The sieve frame shall be 200 mm inside diameter and connecting part between the frame and the wire gauze shall be soldered from inside. A small amount of alcohol or surface - active agent is added as wetting agent. A

*Specification for test sieves (*revised*).

rubber spatula is used for levelling lightly while pouring water on it and allow it to pass through the sieve. Continue pouring of water until the washings are clear. Dry the sieve for 30 minutes in an air oven maintained at 105 to 110°C and allow to cool in a desiccator. Transfer the residue to a tared weighing bottle and weigh accurately.

A-4.3 Calculation

$$\text{Coarse particles, percent by mass} = 100 \times \frac{m}{M}$$

where

m = mass in g of the residue, and

M = mass in g of the material taken for the test.

A-5. HYDROCHLORIC ACID ABSORPTION NUMBER

A-5.1 Reagents

A-5.1.1 Dilute Hydrochloric Acid — 2 N approximately.

A-5.2 Procedure — Weigh accurately about 5 g of the sample into a 300 ml conical flask and add dropwise 40 ml of 2 N hydrochloric acid on the sample from a 50 ml burette graduated in 0.1 ml division. After the flask is stoppered with a rubber stopper, mix them thoroughly by shaking horizontally for about one minute, taking care that the inside wall of the flask shall not be wetted with the hydrochloric acid. Then hold the stoppered flask by the neck and shake it using rotary motion uninterruptedly for a minute till particles start agglomerating.

A-5.3 Stop the shaking when the sample is permeated uniformly with the hydrochloric acid, and scrub off gently the sample adhering to the wall of the flask with a glass rod. This process finishes in about three minutes.

A-5.4 Add dropwise 2 ml of 2N hydrochloric acid and shake thoroughly for about two minutes as mentioned above, so that small particles hit the wall of the flask hard. Add 1 ml of the acid afresh and shake vigorously for about two minutes. Thereafter the amount of dropping acid is decreased gradually (0.5 — 0.3 — 0.2 — 0.1 ml). During the process, the flask may be tapped with palm so that the lumps of the sample adhering to the wall of the flask are removed down.

A-5.5 Continue this operation until the small particles build up to an agglomerate. This test shall be finished in 15 ± 1 minutes from the beginning of the addition of hydrochloric acid.

A-5.6 Calculation — The total amount of 2 N hydrochloric acid required to get to the point of agglomeration shall be regarded as the hydrochloric acid absorption number.

NOTE — During this process, the flask should not be tapped with palm except when the sample particles are adhering to the wall are removed down.

A-6. ACETONE ABSORPTION NUMBER

A-6.0 General — Acetone absorption number is suggested as an alternative to hydrochloric acid absorption number. Handling acetone solution is relatively safer than 2 N hydrochloric acid solution. The flask size recommended is 500 ml. A larger size flask will provide a larger travel for material movement inside the flask for uniform wetting of carbon.

A-6.1 Reagent

A-6.1.1 Test Solution (1 : 9) — Mix 1 part of acetone with 9 parts of water by volume.

A-6.2 Procedure

A-6.2.1 Weigh $5 \pm .01$ g sample of black into a clean dry conical flask. Dispense 10 ml of test solution from burette into flask. Close flask with a rubber stopper. Then shake flask cautiously for one minute using a rotary motion.

A-6.2.2 Open flask and add 5 ml of solution, re-stopper and repeat shaking, as described above, for 15 seconds.

NOTE 1 — As the end point approaches added vigilance is necessary to avoid having the sample pass into a mud stage instead of forming the desired single ball.

NOTE 2 — If the black under test has unusually low absorption value, the indicated solution, addition should be reduced accordingly.

A-6.2.3 Repeat A-6.2.2 until the black agglomerates into small balls. After small balls have formed, add 1 ml of solution in small increments and shake in between addition.

A-6.2.4 When the small balls join to form 2 or 3 larger balls, reduce solution drop by drop followed by vigorous shaking of the flask between each addition.

A-6.3 Calculation — When a single ball is formed that does not break down with continued shaking, record the burette reading. Repeat absorption value as the number of ml of test solution added to the 5 g sample. Duplicate determination should check within ± 1 ml.

A-7. ELECTRICAL RESISTIVITY

A-7.1 Apparatus

A-7.1.1 The apparatus is shown in Fig. 1. Use up to 0.5 V voltmeter and 0.5 A ammeter with 200 graduation mark. It has an electronic box which provides an accurate dc current source, measures resistivity in milliohms and displays it on the digital window, and a screw press for compressing acetylene black to a prefixed packing density (0.70 g/cm^3) at which to measure samples resistivity.

A-7.2 Procedure — Weigh 0.70 g of acetylene black and pour it inside die body with bottom plunger in position. Then insert the top plunger and press it gradually down with hand. Place the entire assembly inside the screw press. Place the 'spacer' provided for attaining fixed packing density between the platen and the base of the screw press. Lower the platen till spacer is securely clamped underneath. Read resistivity in milliohms-cm. The meter reading minus reading of lead resistance in milliohms (obtained by short-circuit without sample) is the resistivity of the acetylene black at 0.70 g/cm^3 packed density in milliohms-cm. The lead resistance is 2 to 3 milliohms as compared to the reading of sample of 70 to 72 milliohms. If the former is ignored, the meter reading is within 3 percent of accuracy. Dividing the reading by 1000, will give resistivity in ohm-cm.

A-7.3 Calculation

$$\text{Electrical resistivity (Ohm-cm)} = \frac{S}{L} \times R$$

where

S = cross-sectional area of the sample in cm^2 ,

L = height of the pressed sample in cm (1 cm), and

R = electrical resistance in ohm of acetylene black sample.

A-8. POUR DENSITY

A-8.1 Procedure — Charge gently up to 100 ml with the sample with a spoon in a tared cylinder (100 ml) in inclined position and weigh to the 0.1 g so that the mass of the sample can be calculated accurately. Stopper the cylinder with a cork stopper and drop it freely fifty times on to a rubber sheet from a height of 5 cm. Calculate the pour density (g/ml) from the volume of the condensed sample.

A-8.2 Calculation

$$\text{A-8.2.1 Pour density (g/ml)} = \frac{M}{V}$$

where

M = mass in g of the sample taken for the test, and

V = volume in ml of the sample after having dropped fifty times.

A-9. APPARENT DENSITY — ALTERNATE METHOD TO POUR DENSITY

A-9.0 General — Apparent density is defined as the mass per unit of external volume. It differs from real density in that part of the unit volume is occupied by voids and not by the material itself. Apparent density, therefore, has a value dependant on the ratio of pore space to substance, being limited in one direction by the real density of the substance itself and in the other by the density of the substance (usually air) occupying the voids. The unit used in grams per cubic centimetre.

A-9.1 Procedure — The method used is to take a container whose internal volume is accurately known and obtain the mass of material required to fill it full, without compressing or packing it. A light plywood body with inside dimensions of 30 cm \times 30 cm is most convenient. The tare mass of the empty box is taken and then it is filled to overflowing. A straight edge is then drawn across the box levelling it off full. The full box is then weighed and the tare mass subtracted to give the mass of carbon for 2700 cm³. Calculate mass per cm³ of carbon.

A-9.2 Calculation — If a container is used having a volume of other than 2700 cm³, its volume must be determined in cm³ (length \times width \times height). The mass in grams required to fill it must be divided by the volume to give the mass per cm³.

A-10. pH VALUE

A-10.1 Weigh 5 g of the sample into a 500 ml beaker and add 150 ml water. Boil it for five minutes and allow to cool to room temperature. Centrifuge it about 8 minutes (2 000 rpm) and decant off the supernatant liquid. Transfer the sludgy residue to a beaker and determine the pH value by means of a glass-electrode pH meter. Determine the pH value three times stirring the sludgy residue with a rod so that it adheres firmly to the electrodes. Repeat the measurement until the values of the three consecutive tests are coincident within the range of 0.1.

APPENDIX B
(*Clause 5.1*)
SAMPLING OF ACETYLENE BLACK

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing samples, the following precautions and directions shall be observed.

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container shall be mixed thoroughly by suitable means.

B-1.5 The sample shall be placed in suitable, clean and dry glass containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost completely filled with the sample.

B-1.7 Each sample container shall be sealed airtight after filling and marked with full details of sampling, date of sampling and year of manufacture of the material.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material drawn from the same batch of manufacture of same grade and the same size shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture or of different sizes of containers, the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

B-2.2 Samples shall be tested for each lot for ascertaining the conformity of the material to the requirements of this specification.

B-2.3 The number of containers (n) to be chosen from a lot shall depend upon the size of the lot (N) and shall be in accordance with Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING
(Clause B-2.3)

Lot Size <i>N</i>	Sample Size <i>n</i>
Up to 50	3
51 to 100	4
101 to 150	5
151 to 300	7
301 and above	10

B-2.4 These containers shall be chosen at random from the lot. In order to ensure the randomness of selection, reference may be made to IS : 4905 - 1968*.

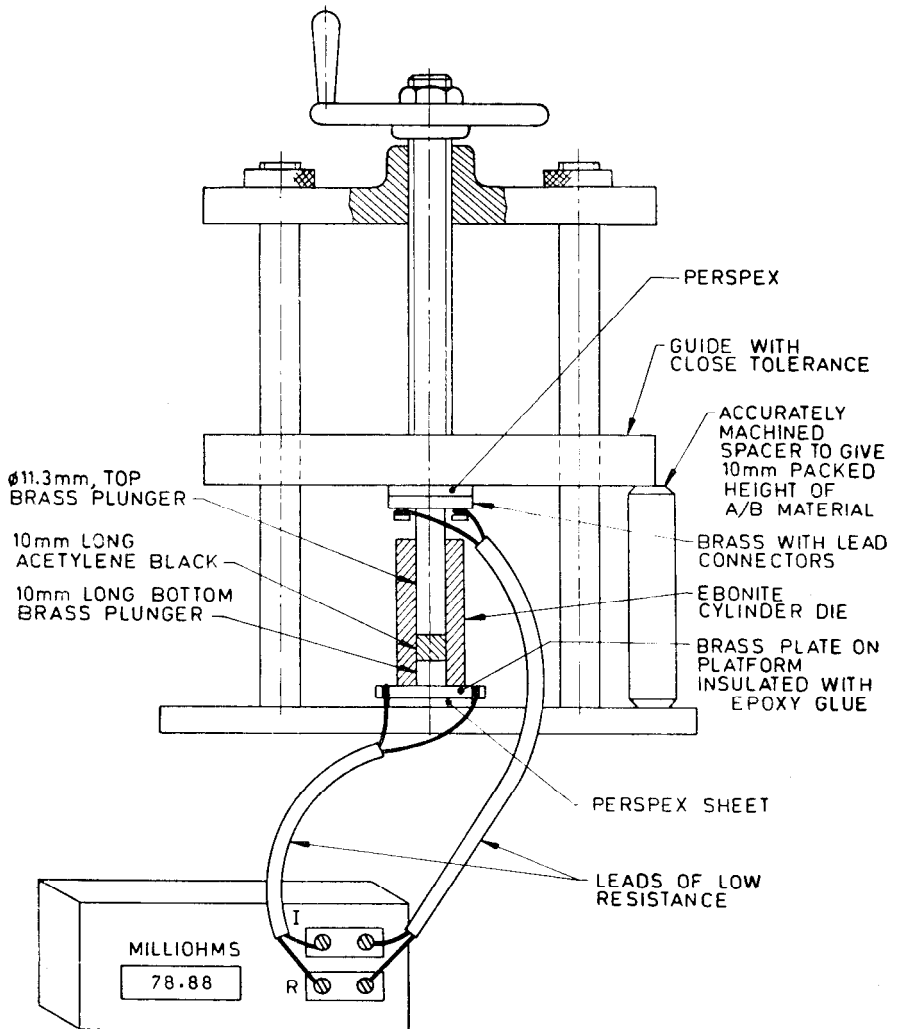
B-3. NUMBER OF TESTS

B-3.1 Test for the determination of all characteristics given in Table 1 shall be conducted on sample.

B-4. CRITERIA FOR CONFORMITY

B-4.1 For declaring the conformity of the lot to the requirements of all characteristics tested on the composite sample, the test result for each characteristic shall satisfy the relevant requirements given in Table 1.

*Methods for random sampling.



NOTE — Note initial milliohm reading with shorting brass plungers top and bottom, this will be between 2 to 5 milliohm in case more then tighten connections.

FIG. 1 ELECTRICAL RESISTIVITY APPARATUS (ACETYLENE BLACK)

(Continued from page 2)

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